JET PROPULSION LABORATORY

INTEROFFICE MEMO
EFC-507-C-XXX
APRIL 22, 1997

To : Mike Sandor
From: Edward Cuddihy
Subject: Chemical Analysis and Outgassing of Plastic Packaged Parts

Ref: ASTM Standard E 595-93, "Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment", Current Edition approved June 15, 1993.

SUMMARY

Four plastic encapsulated microcircuits (PEMs) with the following designations

1) MCR 265
2) 7612382FBA, E24, DA28F016SV, K8055, U6240332
3) AM28F020-15PC, 9618FBB
4) CSI, CAT28FO2OF, I-15, 0955OB

are undergoing qualification testing for spacecraft applications, and were submitted for chemical analysis of their plastic encapsulants, and determination of their outgassing properties. The outgassing test is a requirement imposed on plastic and organic materials intended for use in spacecraft applications. The plastic encapsulants of all four devices were epoxies, and all four passed the "Volatile Condensable Materials" (VCM) outgassing test per NASA/JPL conformance to the referenced ASTM standard. The volatile condensable materials from all four devices were components of epoxy chemistry.

This memo presents a viewpoint that even if a PEM were to fail the outgassing test, the test itself may not be needed as a requirement for PEMs, and therefore could cause unnecessary rejection of otherwise good parts. An alternate purpose for carrying out the outgassing test is discussed.

INTRODUCTION

Four plastic encapsulated microcircuits (PEMs) were submitted for a measurement of their outgassing contents, by a standard volatile condensable materials (VCM) test described in the referenced standard.

JPL's VCM test equipment is located in Bldg. 158, and operated by Phil Stevens, Section 353. The microcircuits were to large to fit intact in the VCM chamber, and it was agreed to cut up the devices to generate small pieces of the plastic encapsulant from each device for the test. Therefore only the plastic packaging material from each device was tested, and none of the other components such as wires, die, etc. were included in the test.

The standard VCM outgassing test proceeds by the following steps:

1) The sample is conditioned by exposure to 50% RH at 25°C for 24 hrs, to generate an absorbed moisture content at a known reference state

2) The conditioned sample is weighed

3) The conditioned sample is then heated for 24 hrs at 125°C, in a vacuum of 1×10^{-5} torr, to cause outgassing of volatile materials, including any absorbed moisture

4) A collector plate positioned near the heated sample is maintained at room temperature, to provide a surface for collection of condensable materials. Non-condensable materials including water vapor discharge into the vacuum system and are not collected

5) After the 24 hr heating period in vacuum, the outgassed sample is recovered and immediately re-weighed in a dry environment, and using the conditioned weight from step 2, yields the <u>total</u> outgassing loss in weight % for the following three volatile components:

   a) absorbed moisture content from conditioning at 50% RH and 25°C for 24 hrs, a non-condensable

   b) all other non-condensable materials

   c) condensable materials, commonly referred to as the "Volatile Condensable Materials" (VCM)

6) The outgassed sample of known dry weight is again conditioned for 24 hrs to 50% RH at 25°C, to allow moisture regain and a measurement of the actual absorbed moisture content in weight % at this reference state. The VCM test report generated by JPL refers to this as "Water Vapor Recovered, WVR, %"

7) The volatile weight loss, not including water vapor, is calculated by subtracting WVR in wt. % from the <u>total</u> loss in wt. % derived in step 5. This weight loss which consists of condensable and all other non-condensable materials, cannot exceed 1.0 wt.% per the referenced ASTM standard.

8) The volatile condensable materials on the collector plate are weighed, to derive the VCM content in weight %, which cannot exceed 0.1 wt. % per the referenced ASTM standard.

The outgassing test yields the the quantity of volatile condensable materials, the quantity of absorbed moisture at 25°C and 50% RH, and the quantity of all other non-condensable materials (step 7 minus step 8), all of which could be used for lot-to-lot comparison of the same PEM to track manufacturing continuity and consistency, or for general comparison with other PEMs and their plastic encapsulants.

EXPERIMENTAL

Four plastic encapsulated microcircuits with the following designations

1) MCR 265
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were submitted for an outgassing test required of organic materials intended for spacecraft applications. The outgassing test and requirements are described in detail in the referenced standard, and JPL's materials laboratory has the equipment to carry out the standardized test.

Not included in the standardized outgassing test is any requirement to have chemical identification of the organic materials undergoing the test, nor to chemically analyze the "Volatile Condensable Materials" which condense on the collector plate. In a discussion amongst ourselves, it was decided to do both of these chemical analyses by "Fourier Transform Infra Red" (FTIR) spectroscopy in JPL's Chemical analysis lab (Mark Anderson).

The FTIR report authored by Mark Anderson is attached. In summary, all four plastic encapsulants were epoxies, and the volatile condensable materials from all four plastic encapsulants were components of epoxy chemistry. There were no surprises. It should be pointed out that the two most common plastic encapsulants in use today are based on either epoxy or polyimide chemistry.

All four plastic encapsulants passed the outgassing test as specified by the referenced ASTM standard, and the Material Labs VCM report is attached. Two VCM runs were made for each plastic encapsulant, and these results and the average are given in the report. The test designator labeled "DEPOSIT" describes the visual appearance of the "Volatile Condensable Materials" (VCM) on the collector plate. One of the four plastic encapsulants had so little VCM that its appearance as a deposit could not be judged.

WISDOM OF THE OUTGASSING TEST

It is the intent of this section to provide an alternate view of the outgassing test that might be more relevant to PEMs as part of flight qualification. Historically, the outgassing test was developed to qualify for space flight applications, plastic and organic materials which in the vacuum of space could outgas volatile materials that could condense on sensitive optical surfaces. Waiving the outgassing test was generally evaluated on the basis that the location of the plastic or organic material in the spacecraft was such that it did or did not pose a risk to sensitive surfaces, even if outgassing occurred.

PEMs most likely will be mounted on printed circuit boards, and overcoated with conformal coating. For outgassing products to depart the plastic encapsulants of PEMs, these volatile materials must first permeate through the conformal coating. Since chemical intuition suggests that the molecular weights of "Volatile Condensable Materials" would be high, permeation of these materials through a conformal coating does not seem to be chemically reasonable. In effect, it can be conjectured that the conformal coating would actually "seal-in" the volatile condensable materials, preventing them from outgassing, and therefore negating any risk.

Using the outgassing test on PEMs therefore, for the exclusive purpose of conforming to a historical spacecraft requirement, may not be relevant for PEMs, and could cause unnecessary rejection of otherwise good parts. It is recommended that Office 507 should consult with Tim O'Donnell of Section 353 on the relevance of the outgassing requirement for PEMs. This author has learned that printed circuit boards, which are made of plastics, are purchased with a specification that the boards meet the requirements of the outgassing test, but I haven't found out who does the test or how it is validated that the boards actually meet the requirement. Perhaps PEMs could be purchased in the same way as boards? In addition, I am informed that assembled boards (populated and conformally coated) are exposed to some thermal-vacuum environment called out by Dr. Dan Taylor also of

Section 353. I have placed a voicemail message with Dan for information. Perhaps Dan's thermal-vacuum exposure may be sufficient for conformally coated PEMs?

Perhaps the real wisdom of doing the outgassing test as well as FTIR on qualification parts, which are simple tests, is to derive a record of the chemical signature and fingerprints of the encapsulant chemistry and condensable outgassing products, as it reflects the current manufacturing state. Given that the parts become qualified for flight, then part of any future DPA on lots purchased for actual flight, would be to include outgassing and FTIR for comparison with the qualification parts. Changes in encapsulant chemistry, VCM chemistry, and quantities of both condensable and non-condensable materials, could be an indicator of manufacturing changes which may not be qualified, and represent a potential problem.

In addition, the VCM test provides as a bonus the quantity of absorbed moisture at 50% RH and 25°C. Since "popcorning" is related to the quantity of absorbed moisture in the plastic encapsulant, the VCM test result can be viewed as a measurable index relative to "popcorning". If it can be assumed that the parts would be stored at nominal room conditions of 50% RH and 25°C, then the absorbed moisture content reported by the VCM test would also be that quantity of absorbed water in the part when soldered on a printed circuit board, as for example, during a high temperature wave-soldering process. If no "popcorning" occurs with the qualification parts, then that quantity of absorbed moisture could be judged as no risk, and desired in future lots. Any significant variations in moisture content from lot-to-lot at 25°C and 50% RH could be the result of manufacturing changes which may represent a potential problem.

"Popcorning" is often studied by intentionally increasing the absorbed moisture content of the PEM, above that which would normally be in equilibrium with the ambient environment. One common approach is to expose the PEM to 85°C/85%RH, to drive up the absorbed moisture content. From hygroscopic principles, it can be roughly estimated that the absorbed moisture content from exposure to 85°C/85%RH could be about 10 times higher than that from exposure to 25°C/50%RH. Thus if a PEM resists "popcorning" from exposure to 85°C/85%RH, than a ten-fold reduction in absorbed moisture content from storage at 25°C/50%RH should represent a safe reliability margin.

It can be suggested that the real wisdom of the outgassing test is not to meet a potentially non-applicable spacecraft requirement, but to

a) generate a chemical signature of the qualification part useful to track manufacturing continuity and consistency, and to

b) measure an absorbed moisture content at a known and practical storage environment, as an index relative to "popcorning"

JPL MATERIALS LABORATORY VCM REPORT

SECTION 355

REQUEST NO. 97270/1

TO: E.CUDDIHY	BLDG. 303	EXT. 4-3188	DATE 2-21-97
FROM: P.STEVENS			EXT. 4-0775
SUBJECT: MICRO VCM			

VCM ANALYSES WERE PERFORMED ON FOUR PLASTIC ELECTRONIC PACKAGES. COLLECTED CONDENSABLES WERE ANALYZED BY THE CHEMISTRY LABORATORY.

MATERIAL	MCR 265			7612382FBA, E24, DA28F016SV,K8055, U6240332.			AM28F020-150PC, 9618FBB.			CSI,CAT28F020F, 1-15 09550B.		
BATCH NO.	A.A.			A.A.			A.A.			A.A.		
MANUFACTURER	A.A.			A.A.			A.A.			A.A.		
MIX RATIO	A.A.			A.A.			A.A.			A.A.		
CURE	A.A.			A.A.			A.A.			A.A.		
SAMPLE NO.	5	6	AVGE	7	8	AVGE	9	10	AVGE	11	24	AVGE
WT. LOSS %	0.45	0.46	0.45	0.23	0.22	0.22	0.41	0.45	0.43	0.40	0.41	0.40
WATER VAPOR RECOVERED, WVR, %	0.28	0.25	0.26	0.14	0.11	0.12	0.19	0.17	0.18	0.21	0.18	0.19
(WT, LOSS - WVR) %	0.17	0.21	0.19	0.09	0.11	0.10	0.22	0.28	0.25	0.19	0.23	0.21
VCM %	0.04	0.08	0.06	0.02	0.01	0.01	0.03	0.05	0.04	0.04	0.04	0.04
DEPOSIT	OPAQUE			NEGLIGIBLE			OPAQUE			OPAQUE		

PREPARED BY	APPROVED BY
	P Stevens P.STEVENS.

REQUESTER

JPL 3965 R 7/90

cc:Mail for: Edward F Cuddihy

Subject: Beefed Up Report
From: Mark S Anderson at jpl-serac 4/21/97 3:22 PM
To: Edward F Cuddihy at JPL-514PO

JPL ANALYTICAL CHEMISTRY LABORATORY H041

To: Ed Cuddihy 3/3/97

From: Mark S. Anderson

Subject: Electronic Packaging Materials

1. MCR 265
2. 7612382FBA, E24, DA28F016SV, K8055, U6240332
3. AM28F020-15PC, 9618FBB
4. CSI, CAT28F020F, I-15, 09550B

Purpose:

Electronic packaging materials were submitted for chemical analysis of the encasing material and the Vacuum Condensable Materials (VCM) residue. VCM is a standard test to measure outgassed material that can condense on sensitive surfaces. The chemical characterization of the VCM aids in the assessment of the molecular contamination potential of the parts. The data is also useful for having a chemical fingerprint record of the materials for future materials analysis.

Method:

The materials and residue were analyzed using an Fourier Transform Infrared (FTIR) spectroscopy using the Diffuse Reflectance (DRIFT) technique. FTIR provides chemical functional group information for quantitative analysis and qualitative identification of materials. The DRIFT spectra are attached with little or no data manipulation. Phil Stevens (353) ran the VCM test. The VCM table is attached.

Results:

The encasing material for all of the parts is epoxy based. These are common, standard epoxies that are filled with glass. The #2 sample has an anhydride cured epoxy the others are amine cured.

The VCM (see P. Stevens attached report) levels are in the low range and for most situations do not pose a contamination concern. The VCM residue for the #1 part is mainly epoxy (Bis Phenol A component) that is from a slight excess of the part A component. The #2 VCM has mainly Aliphatic Hydrocarbon which this is a common trace contaminant. The #3 and #4 parts have a VCM that is a mixture of Aliphatic Hydrocarbon and Silicone. Silicones are also a common trace contaminant.

JPL **MATERIALS LABORATORY VCM REPORT**

SECTION 355

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MATERIAL	MCR 265			7612382FBA, E24, DA28F016SV,K8055, U6240332.			AM28F020-150PC, 9618FBB.			CSI,CAT28F020F, 1-15 09550B.		
BATCH NO.	A.A.			A.A.			A.A.			A.A.		
MANUFACTURER	A.A.			A.A.			A.A.			A.A.		
MIX RATIO	A.A.			A.A.			A.A.			A.A.		
CURE	A.A.			A.A.			A.A.			A.A.		
SAMPLE NO.	5	6	AVGE	7	8	AVGE	9	10	AVGE	11	24	AVGE
WT. LOSS %	0.45	0.46	0.45	0.23	0.22	0.22	0.41	0.45	0.43	0.40	0.41	0.40
WATER VAPOR RECOVERED, WVR, %	0.28	0.25	0.26	0.14	0.11	0.12	0.19	0.17	0.18	0.21	0.18	0.19
(WT, LOSS - WVR) %	0.17	0.21	0.19	0.09	0.11	0.10	0.22	0.28	0.25	0.19	0.23	0.21
VCM %	0.04	0.08	0.06	0.02	0.01	0.01	0.03	0.05	0.04	0.04	0.04	0.04
DEPOSIT	OPAQUE			NEGLIGIBLE			OPAQUE			OPAQUE		
FTIR Analysis of VCM residue	Epoxy			Trace, Aliphatic Hydrocarbon			Aliphatic hydrocarbon and silicone			Aliphatic hydrocarbon and silicone		

C:\D97\ECFel-4, ECMRI-4.BSP

P Stevens

PREPARED BY	APPROVED BY P.STEVENS.

REQUESTER

JPL 3965 R 7/90

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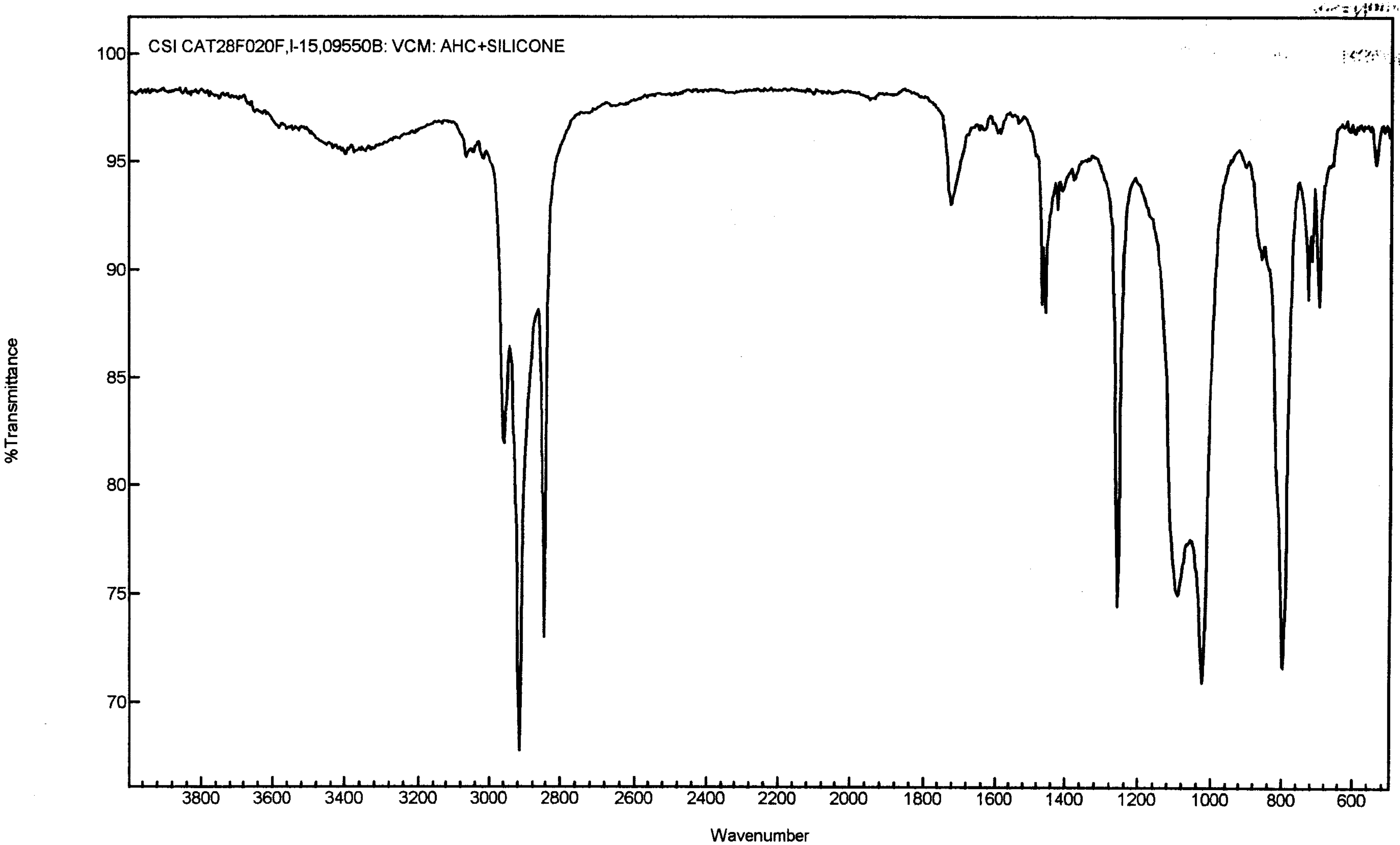
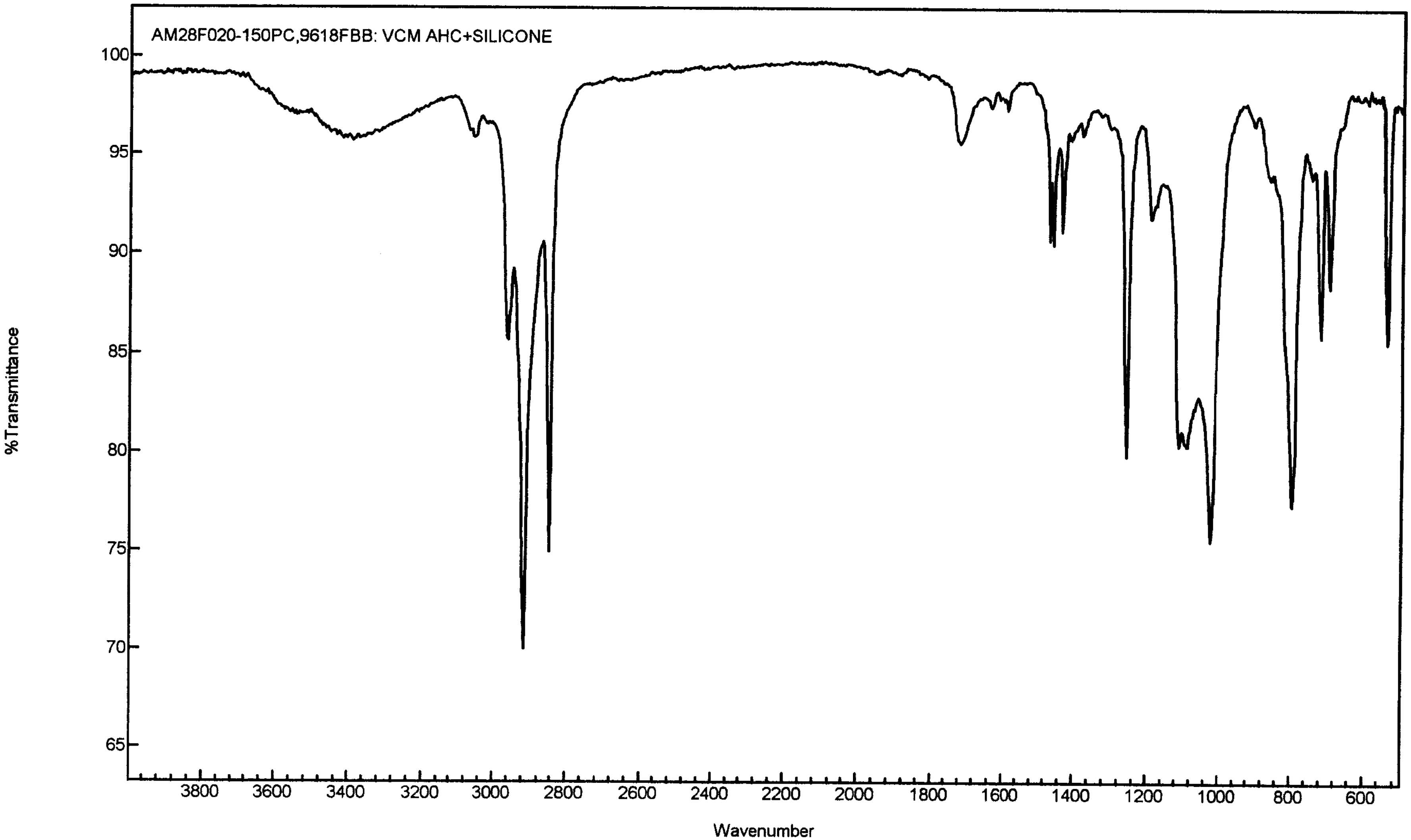
JPL 1140-1 6/84

FTIR: JPL Analytical Chemistry



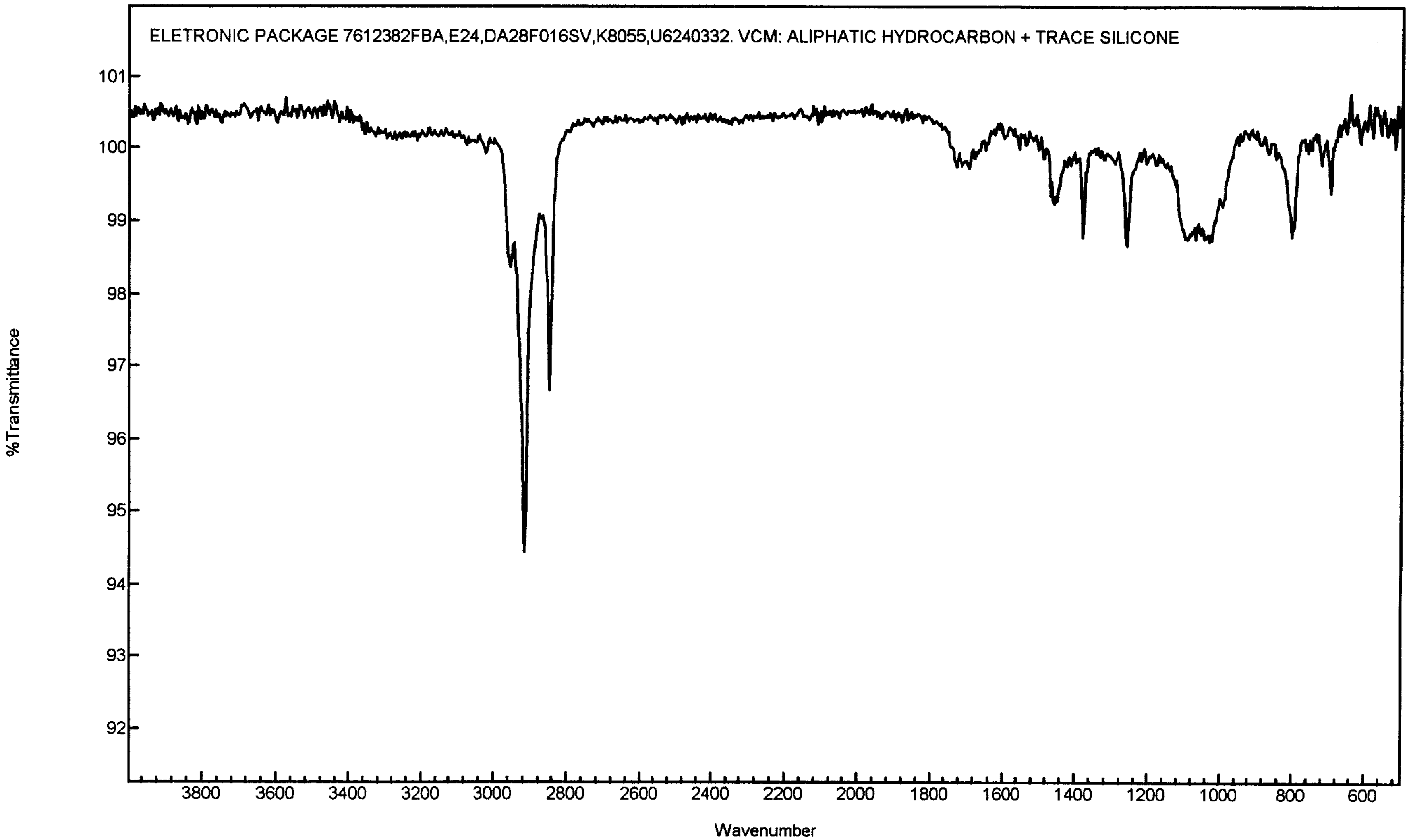
Name
CSI CAT28F020F,I-15,09550B: VCM: AHC+SILICONE

FTIR: JPL Analytical Chemistry



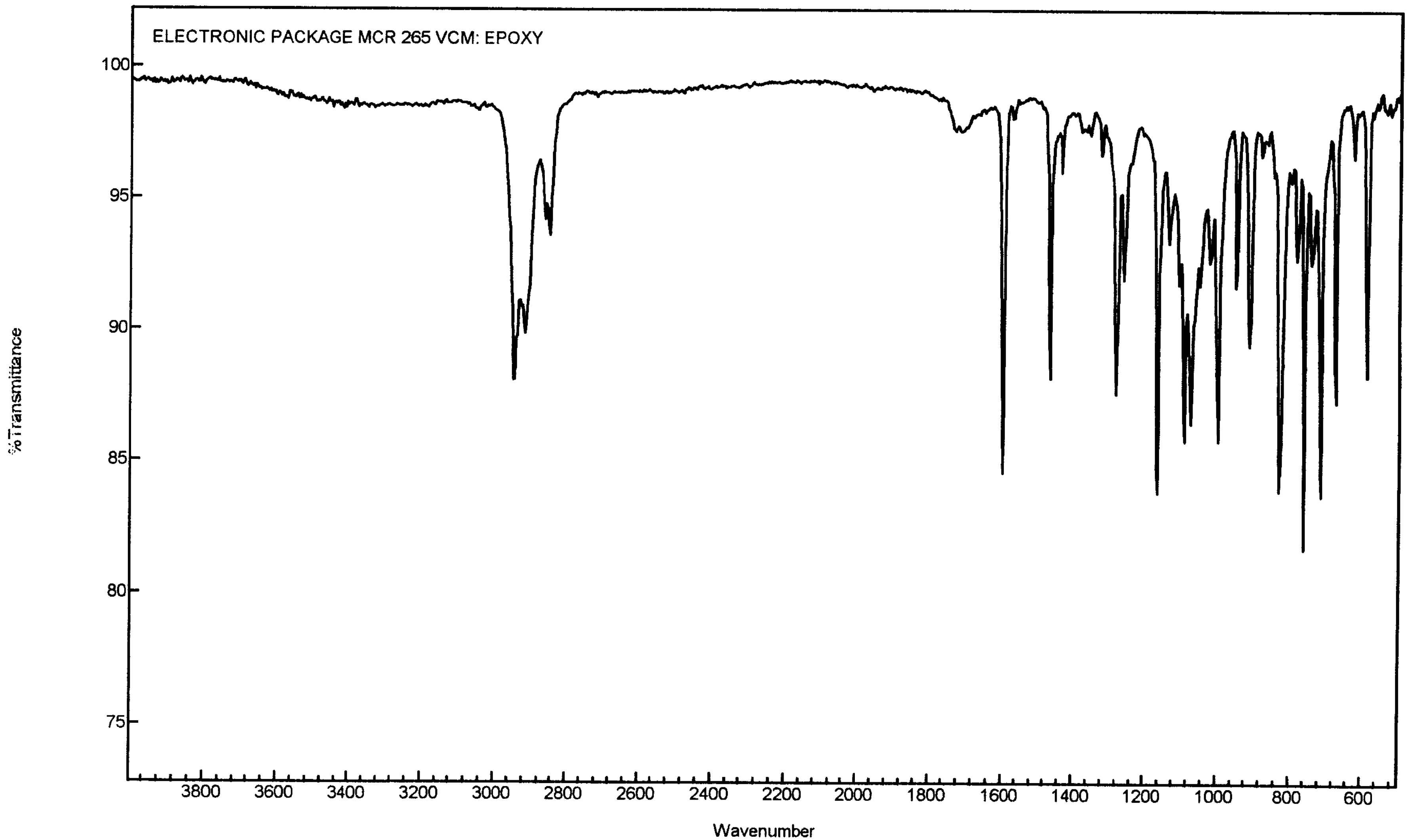
Name
AM28F020-150PC,9618FBB: VCM AHC+SILICONE

FTIR: JPL Analytical Chemistry



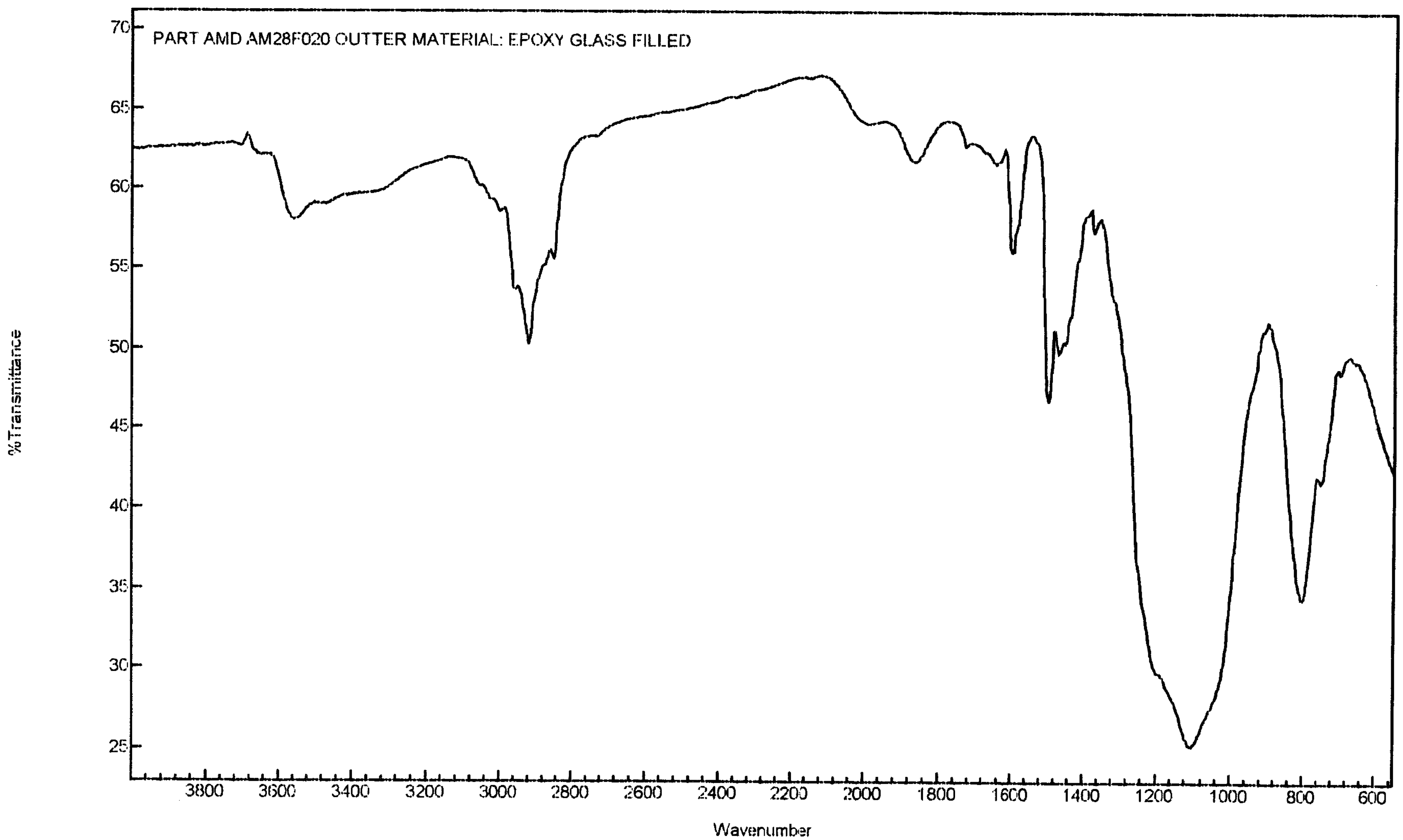
Name
ELETRONIC PACKAGE 7612382FBA,E24,DA28F016SV,K8055,U6240332. VCM: ALIPHATIC HYDROCARBON + TRACE SILICONE

FTIR: JPL Analytical Chemistry



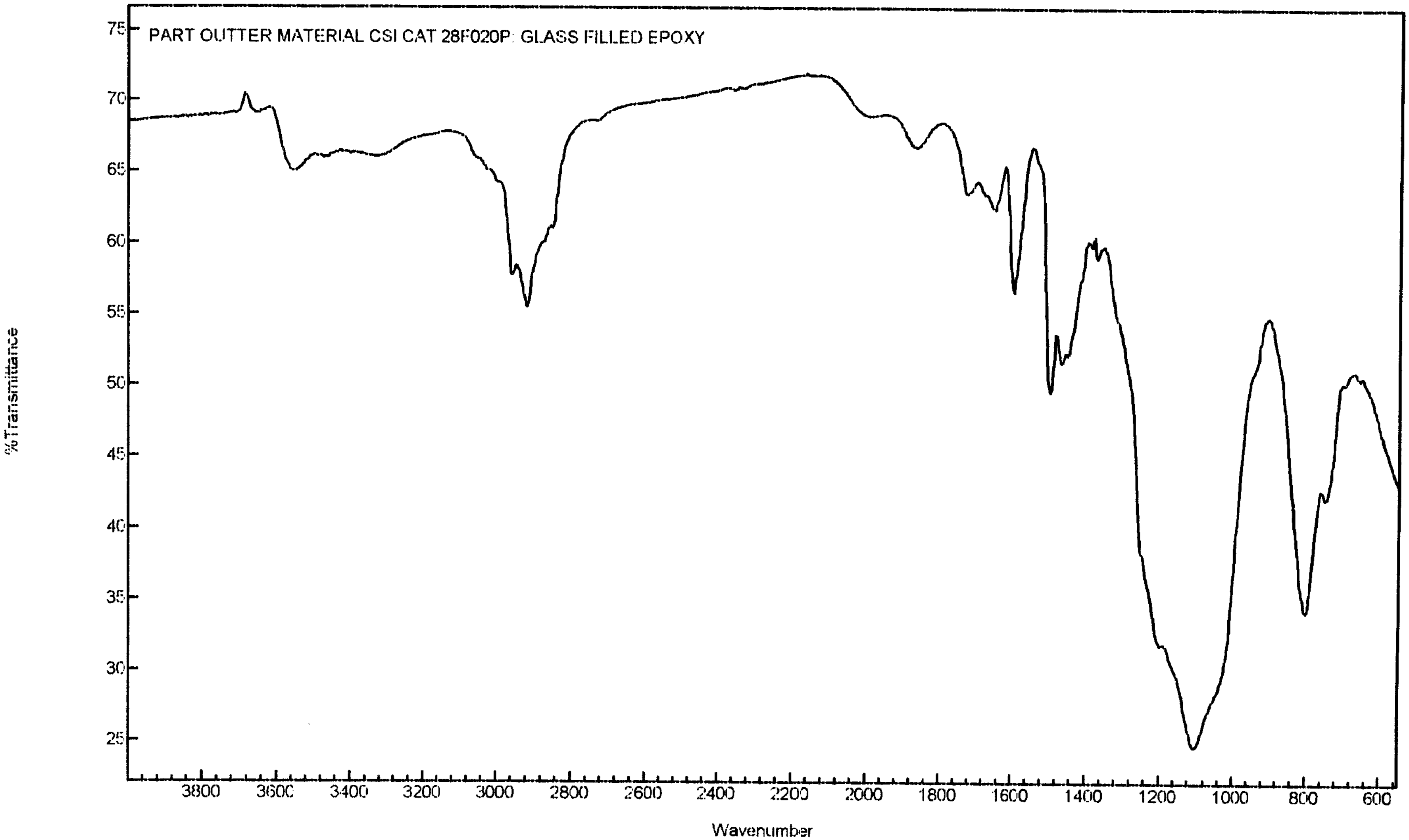
Name
ELECTRONIC PACKAGE MCR 265 VCM: EPOXY

FTIR: JPL Analytical Chemistry



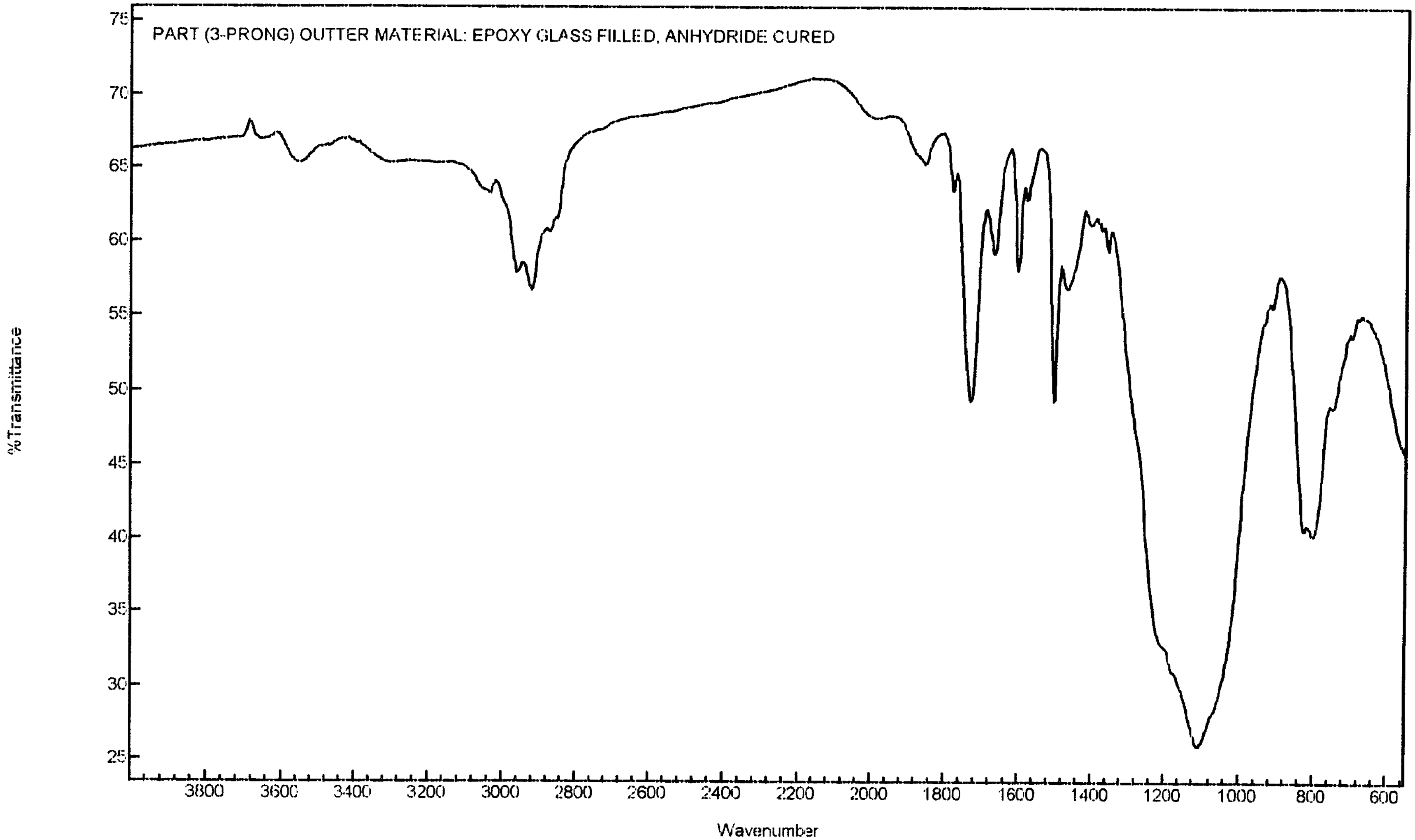
Name
PART AMD AM28F020 OUTTER MATERIAL: EPOXY GLASS FILLED

FTIR: JPL Analytical Chemistry



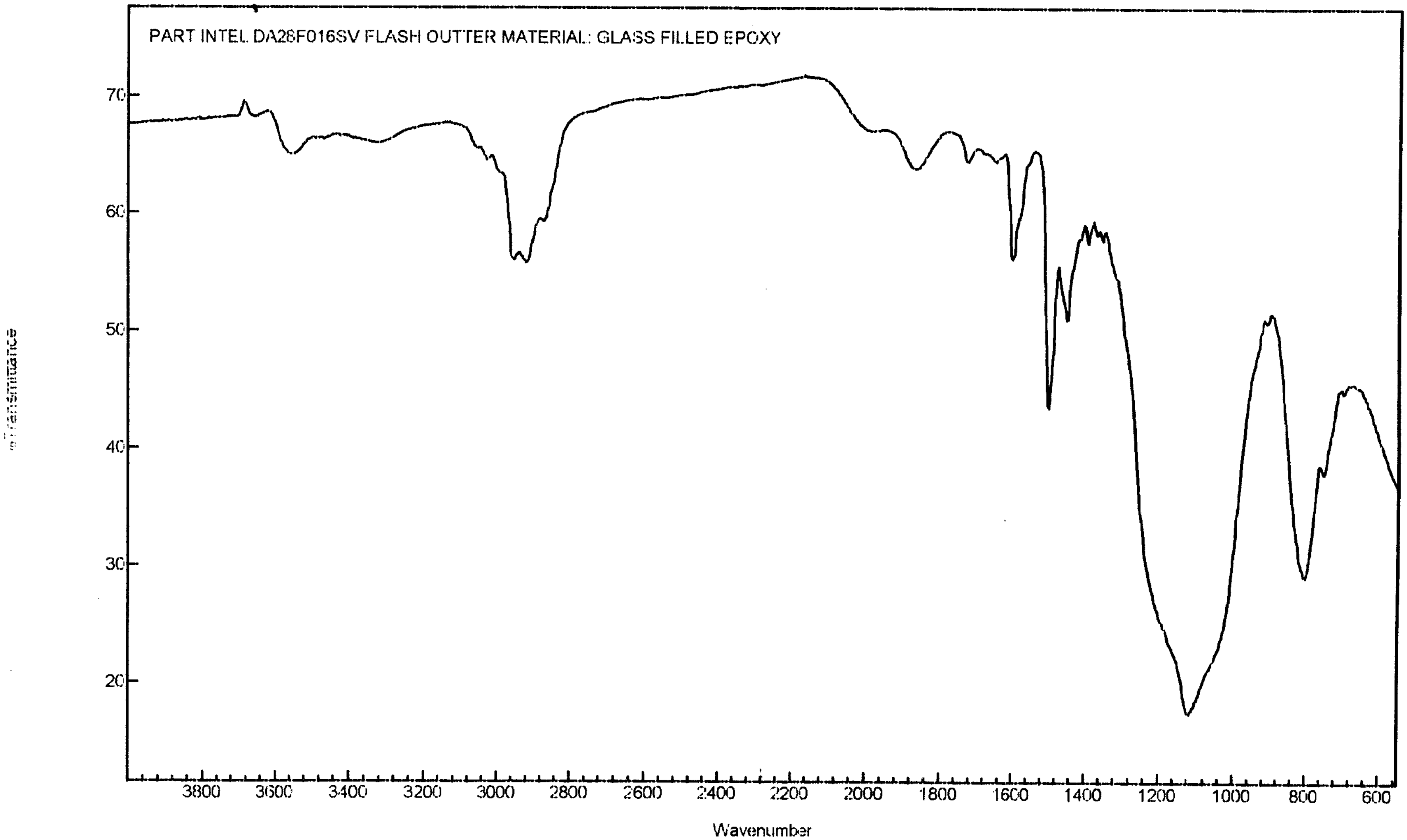
Name
PART OUTTER MATERIAL CSI CAT 28F020P: GLASS FILLED EPOXY

FTIR: JPL Analytical Chemistry



Name
PART (3-PRONG) OUTTER MATERIAL: EPOXY GLASS FILLED, ANHYDRIDE CURED

FTIR: JPL Analytical Chemistry



Name
PART INTEL DA28F016SV FLASH OUTTER MATERIAL: GLASS FILLED EPOXY